भारतीय मानक
Indian Standard

IS 2632 : 2023

क्रोटोनल्डिहाइड — विशिष्टि

(पहला पुनरीक्षण)

Crotonaldehyde — Specification

(First Revision)

ICS 71.080.80

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भारतीय मानक ब्यूरो
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January 2023 **Price Group 7**

Organic Chemicals, Alcohols and Allied Products Sectional Committee, PCD 09

FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Organic Chemicals, Alcohols and Allied Products Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

Crotonaldehyde is a useful chemical intermediate by virtue of its highly reactive character. It undergoes the characteristic reactions of the ethylenic double bond as well as those of simple aldehydes. It is a starting material for a wide range of aldehyde-amine type of rubber antioxidants and accelerators; for example, crotonic acid, butyraldehyde and butanol, quinoline yellow and other dyes and for certain synthetic resins. It is also used as a denaturant for industrial alcohol. The production of crotonaldehyde has been recently established in the country. This standard is expected to assist manufacturers in regulating the quality of material produced and the consumers in selecting a material of acceptable quality for use as a denaturant for industrial alcohol.

This standard was first published in 1964. In this (first revision), two grades have been formed, Grade 1 (99 percent assay), an unsaturated aldehyde with commercial applications in the fragrance, food, paints, and agriculture and adhesive industries and Grade 2 (85 percent assay), used as alcohol denaturant. Characteristics of acidity (as crotonic acid) and water content are incorporated in Table 1. Gas chromatography method to determine the purity level is also incorporated for both the grades as alternate method to chemical method.

This standard is intended chiefly to cover the technical provisions relating to the supply of crotonaldehyde.

The composition of the Committee, responsible for the formulation of this standard is listed in Annex E.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 2022 'Rules for rounding off numerical values (*second revision*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

CROTONALDEHYDE — SPECIFICATION

(First Revision)

1 SCOPE

This standard prescribes the requirements, the methods of sampling and testing for crotonaldehyde.

2 REFERENCES

The following standards contain provisions, which through reference in this text constitute the provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most editions of the standards indicated below:

IS No.	*Title*
IS 323 : 2009	Rectified spirit for industrial use — Specification (*second revision*)
IS 1070 : 1992	Reagent grade water — Specification (*third revision*)

3 GRADES

The material shall be of the following grades:

a) Grade 1, purity 99 percent.
b) Grade 2, purity 85 percent.

4 REQUIREMENTS

4.1 Description

The material shall be a clear liquid with a pungent suffocating odour consisting essentially of crotonaldehyde. The material shall be free from sediment and suspended matter.

4.2 The material shall also comply with the requirements specified in Table 1, when tested according to the methods prescribed in col 5 and col 6 of the Table 1.

Table 1 Requirements for Crotonaldehyde
(*Clauses* 4.2 *and* 7.1)

Sl No.	**Characteristic**	**Requirement**		**Method of test, Ref to**	
		Grade 1	Grade 2	Annex	IS
(1)	(2)	(3)	(4)	(5)	(6)
i)	Specific gravity at 27 °C/27 °C, *Max*	0.860	0.872	A	—
ii)	Aldehyde content (as crotonaldehyde), percent by weight, *Min*	99	85	B	—
iii)	Acidity (as acetic acid), percent by weight, *Max*	—	0.3	C	—
iv)	Acidity (as crotonic acid), percent by weight, *Max*	0.3	—	C	—
v)	Water content, percent by weight, *Max*	0.2	—	—	IS 2362

5 PACKING AND MARKING

5.1 Packing

5.1.1 The material shall be packed in suitable containers as agreed to between the purchaser and the supplier and subject to the provisions of applicable Red Tariff No. for Rules and Rates for conveyance by rail of explosives and other dangerous goods, issued by the Indian Railway Conference Association with any alterations or additions made thereafter.

5.1.2 All containers in which the material is packed shall be dry and clean so that no impurities harmful to the end use of the material are introduced.

5.2 Marking

5.2.1 The material shall be supplied in accordance with the marking and delivery instructions given by the purchaser.

5.2.2 Each container shall be marked legibly and indelibly with the following information:

a) Name of the material;
b) Name of manufacturer and recognized trade-mark, if any;
c) Net, gross and tare weight;
d) Month and year of manufacture;
e) Batch or lot number (if applicable); and
f) Any other statutory requirements.

5.2.3 *BIS Certification Marking*

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the *Bureau of Indian Standards Act*, 2016 and the Rules and Regulations framed thereunder, and the products may be marked with the Standard Mark.

6 SAMPLING

6.1 Preparation of Test Samples

The method of drawing representative samples of the material shall be as prescribed in Annex D.

6.2 Number of Tests

Tests for the determination of all the characteristics specified shall be conducted on the composite sample.

6.3 Criteria for Conformity

The material shall be taken as conforming to the specification, if the test results of the composite test sample satisfy the corresponding requirements given in **4** and Table 1.

7 TEST METHODS

7.1 Tests shall be conducted as prescribed in col 5 and col 6 of Table 1 respectively.

7.2 Quality of Reagents

Unless specified otherwise, ‘pure chemicals’ and distilled water (*see* IS 1070) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

ANNEX A

[*Table* 1, *Sl No.* (i)]

DETERMINATION OF SPECIFIC GRAVITY

A-1 APPARATUS

A-1.1 Specific Gravity Bottle, 50 m1 capacity of regnault type or pyknometer of 25 ml capacity.

A-2 PROCEDURE

Clean, dry and weigh the specific gravity bottle and the stopper or pyknometer. Fill the bottle with water and immerse it up to the neck in a constant temperature bath maintained at (27.0 ± 0.2) °C for 20 min and keep at this temperature for additional 20 min. Suck off the water with a bit of filter paper till the level reaches the graduation mark and weigh again. Empty the bottle, clean and dry. Repeat the operation with the material at 27 °C.

A-3 CALCULATION

Specific gravity at 27 °C/27 °C = $\frac{A-B}{C-B}$

where

A = weight of the specific gravity bottle with the material at 27 °C, g;

B = weight of the specific gravity bottle, g; and

C = weight of the specific gravity bottle with water at 27 °C, g.

ANNEX B

[*Table* 1, *Sl No.* (ii)]

DETERMINATION OF CROTONALDEHYDE CONTENT

B-1 CHEMICAL METHOD (FOR BOTH GRADES)

B-1.1 Reagents

B-1.1.1 *Alcoholic Solution of Hydroxylamine Hydrochloride,* 0.5 N

Dissolve 35 g of hydroxylamine hydrochloride in 150 ml of distilled water and dilute to 1 litre with methanol.

B-1.1.2 *Aqueous Solution of Triethanolamine,* 0.5 N

Transfer 65 ml (74 g) of 98 percent triethanolamine to a 1 000 ml volumetric flask and dilute to the mark with water.

B-1.1.3 *Standard Sulphuric Acid Solution (Dilute),* 0.5 N.

B-1.1.4 *Bromophenol Blue Indicator,* 0.04 percent (*w/v*) alcoholic solution.

B-1.2 Procedure

B-1.2.1 *Preparation of Neutral Alcoholic Solution of Hydroxylamine Hydrochloride*

Add 30 ml of alcoholic solution of bromophenol blue indicator to 1 litre of the hydroxylamine hydrochloride reagent. From a burette add triethanolamine solution until the solution appears greenish-blue by transmitted light. Prepare the solution fresh before each series of the analysis.

B-1.2.2 Prepare a sufficient number of heat-resistant pressure bottles to make all the blank and sample determinations in duplicate. To each bottle add 65 ml of neutralized alcoholic solution of hydroxylamine hydrochloride and from a burette add exactly 50 ml of triethanolamine solution. Before capping, purge the bottle for 2 min with a gentle stream of nitrogen. This is best accomplished by means of a glass tube inserted through the neck of the bottle and clamped so that the opening is just above the surface of the liquid. Reserve two bottles for blanks and into each of the other bottles introduce about one gram of the sample weighed to the nearest 0.1 mg. Allow the samples to stand together with the blanks at room temperature for 60 min. Shake the bottles occasionally. Cool each of the bottle slightly with tap water and uncap carefully to prevent loss of contents. Allow the contents to return to room temperature and titrate each of the blanks with standardized dilute sulphuric acid to greenish-blue end-point. Titrate each of the samples with standardized dilute sulphuric acid to the colour of the blanks approaching the end point dropwise until the colours match by transmitted light.

B-1.3 Calculation

Aldehyde content (as crotonaldehyde), percent by weight = $\frac{(B-A) \times 7.00 \times N}{W}$

where

B = average volume of standard sulphuric acid solution required for the blanks, ml;

A = volume of standard sulphuric acid solution required for the sample, ml;
N = normality of standard sulphuric acid solution; and
W = weight of sample taken for the test, g.

B-2 GAS CHROMATOGRAPHIC METHOD (FOR GRADE 1)

B-2.1 General

A representative sample is introduced into a gas chromatograph equipped with 6 percent cyanopropylphenyl, 94 percent dimethylpolysiloxane bonded phase fused silica capillary column. Suitable carrier gas transports the vaporized sample through the column where the components are separated by the chromatographic process. Components are sensed by a flame ionization detector as they elute from the column. The detector signal is processed by an electronic data acquisition system. The product and other components are identified by comparing their retention times to the ones identified by analyzing standards under identical conditions. The concentration of all components are determined in mass percent area by area normalization of the peak areas.

B-2.2 Apparatus

B-2.2.1 *Gas Chromatograph*

Any gas liquid chromatograph equipped with split/spilt less injector and FID detector can be used with following accessories and operating conditions. A heated flash vaporizing injector designed to provide a linear sample spilt injection (for example, 2:1) is required for proper sample introduction. Carrier gas controls shall be of adequate precision to provide reproducible column flows and split ratios in order to maintain analytical integrity. Pressure control devices and gauges shall be designed to attain the linear velocity required in the column used. A hydrogen flame ionization detector with associated gas controls and electronics, designed for optimum response with open tabular columns, is required.

B-2.2.2 *Sample Introduction*

Manual or automatic liquid syringe sample injection to the splitting injector is employed. Devices capable of 0.1 µl to 0.5 µl injections are suitable. It should be noted that inadequate splitter design, poor injection technique, and overloading the column can result in poor resolution.

B-2.2.3 *Column*

This test method utilizes a fused silica open tubular capillary column coated with 6 percent cyanopropylphenyl, 94 percent dimethylpolysiloxane of 30 m in length, 0.53 mm or suitable internal diameter and film thickness 3.0 µm or equivalent.

B-2.2.4 *Typical Instrument Condition*

Detector	:	Flame Ionization detector (FID)
Injector Temperature	:	240 °C
Detector Temperature	:	240 °C
Oven Temperature program		
Initial Temperature	:	60 °C
Initial Hold Time	:	0 min
Ramp-1	:	12 °C/min
Final Temperature	:	240 °C
Total run time	:	15 min
Carrier Gas	:	Nitrogen/Helium (3 ml/min or suitable as per column ID)

B-2.2.5 *Data Acquisition System*

Any suitable data integrator or PC based GC software which can handle features like external/internal standard calculations, etc.

B-2.3 Reagent

B-2.3.1 *Crotonaldehyde,* pure AR grade

B-2.4 Identification

B-2.4.1 *Identification*

Determine the retention time of product by injecting small amount of highly pure material either individually or synthetic blend mixture.

B-2.4.2 *Analysis Procedure*

Inject 1 µl of sample without any air bubble trapped in the syringe. Determine the mass concentration of all products by area normalization method.

Calculate the assay of crotonaldehyde on dry basis as below:

$$\text{Assay} = \text{Crotonaldehyde by GC} - \left(\frac{\text{Water x Crotonaldehyde by GC}}{100} + \frac{\text{Acidity x Crotonaldehyde by GC}}{100}\right)$$

B-3 GAS CHROMATOGRAPHIC METHOD (FOR GRADE 2)

B-3.1 General

A representative sample is introduced into a gas chromatograph equipped with a porous polymer adsorbent packed column. Suitable carrier gas transports the vaporized sample through the column where the components are separated by the chromatographic process. Components are sensed by a thermal conductivity detector as they elute from the column. The detector signal is processed by an electronic data acquisition system. The product and other components are identified by comparing their retention times to the ones identified by analyzing standards under identical conditions. The concentration of all components are determined in mass percent area by area normalization of the peak areas.

B-3.2 Apparatus

B-3.2.1 *Gas Chromatograph*

Any gas liquid chromatograph equipped with direct injector and thermal conductivity detector (TCD) can be used. Carrier gas controls shall be of adequate precision to provide reproducible column flows. Pressure control devices and gauges shall be designed to attain the linear velocity required in the column used. A thermal conductivity detector with associated gas controls and electronics, designed for optimum response with packed columns, is required.

B-3.2.2 *Sample Introduction*

Manual or automatic liquid syringe sample injection to the injector is employed. Devices capable of 0.5 µl to 1.0 µl injections are suitable. It should be noted that poor injection technique and overloading the column can result in poor resolution.

B-3.2.3 *Column*

This test method utilizes a packed (SS or glass) column with 3 m in length, 3.18 mm outer diameter, or suitable length.

NOTE — Porapak Q is an ethylvinylbenzene and divinylbenzene copolymer bead which is used in packed column gas chromatography for separating light gases and volatiles.

B-3.2.4 *Typical Instrument Condition*

Detector	:	Thermal Conductivity Detector (TCD)
Injector Temperature	:	240 °C
Detector Temperature	:	240 °C
Oven Temperature program		
Initial Temperature	:	160 °C
Initial Hold Time	:	0 min
Ramp 1	:	8 °C / min
Final Temperature	:	240 °C
Hold Time	:	6 min
Total run time	:	16 min
Carrier Gas	:	Hydrogen (30 ml/min)

B-3.2.5 *Data Acquisition System*

Any suitable data integrator or PC based GC software which can handle features like external/internal standard calculations, etc.

B-3.3 Reagent

B-3.3.1 *Crotonaldehyde,* pure AR Grade

B-3.4 Identification

B-3.4.1 *Identification*

Determine the retention time of product by injecting small amount of highly pure material either individually or synthetic blend mixture.

B-3.4.2 *Analysis Procedure*

Inject 1 µl of sample without any air bubble trapped in the syringe. Determine the mass concentration of product by area normalization method.

Calculate the assay of crotonaldehyde on dry basis as below:

$$\text{Assay} = \text{Crotonaldehyde by GC} - \left(\frac{\text{Acidity x Crotonaldehyde by GC}}{100}\right)$$

NOTE — The above gas chromatographic conditions are suggestive. However any GC having different columns (packed/Capillary having different length/diameter/film thickness) and different carrier gas (He, H_2 or N_2), with different calibration technique (internal standard, external standard, area normalization) may be used provided standardization/calibrations are done after setting up chromatographic conditions for required resolution.

ANNEX C
DETERMINATION OF ACIDITY

[*Table* 1, *Sl No.* (iii) *and* (iv)]

C-1 REAGENTS

C-1.1 Rectified Spirit, 95 percent (*see* IS 323).

C-1.2 Standard Sodium Hydroxide Solution, less than equal to 0.1 N.

C-1.3 Phenolphthalein indicator

Dissolve 0.5 g of phenolphthalein in 60 ml of rectified spirit diluted with water to 100 ml. Make the solution faintly pink by adding dilute sodium hydroxide solution.

C-2 PROCEDURE

Depending on the degree of acidity, chooses a suitable quantity of the material. Dissolve the material in 50 ml of rectified spirit, neutralized to phenolphthalein and titrate the mixture with standard sodium hydroxide solution using 0.5 ml of the phenolphthalein indicator.

C-3 CALCULATION

C-3.1 Calculation for Grade 1

Acidity (as crotonic acid), percent by weight

$$= \frac{8.6 \text{ x } V \text{ x } N}{W}$$

where

V = volume of standard sodium hydroxide solution, ml;
N = normality of standard sodium hydroxide solution; and
W = weight of the material taken for the test, g.

C-3.2 Calculation for Grade 2

Acidity (as acetic acid), percent by weight $= \frac{6 \text{ x } V \text{ x } N}{W}$

where

V = volume of standard sodium hydroxide solution, ml;
N = normality of standard sodium hydroxide solution; and
W = weight of the material taken for the test, g.

ANNEX D

(*Clause* 4.1)

SAMPLING OF CROTONALDEHYDE

D-1 GENERAL REQUIREMENTS OF SAMPLING

D-1.1 In drawing, preparing, storing and handling test samples, the following precautions and directions shall be observed.

D-1.2 Samples shall be taken in a protected area with good ventilation.

D-1.3 Sampling instrument shall be clean and dry.

D-1.4 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

D-1.5 To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by shaking, stirring, rolling, circulation of storage tank or by any other suitable means.

D-1.6 The samples shall be placed in suitable, clean, dry and air-tight, amber-coloured glass or metal containers on which the material has no action.

D-1.7 The sample containers shall be of such a size that they are almost completely filled by the sample.

D-1.8 Each sample container shall be seated air-tight with a suitable stopper after filling and marked with the manufacturer's name or trademark, the month and year of manufacture of the material, the batch number (if available), and other details of sampling, such as the date of sampling, sampler's name, etc.

D-2 SAMPLING INSTRUMENT

D-2.1 It is made of thick glass or metal on which the material has no action and is 20 mm to 40 mm in diameter and 400 mm to 800 mm in length (*see* Fig. 1). The upper and lower ends are conical and reach 5 mm to 10 mm diameter at the narrow ends. Handling is facilitated by two rings at the upper end. For drawing samples, the apparatus is first closed at the top with the thumb or a stopper and lowered till a desired depth is reached. It is then opened for a short time to admit the material at the desired depth and finally closed and withdrawn.

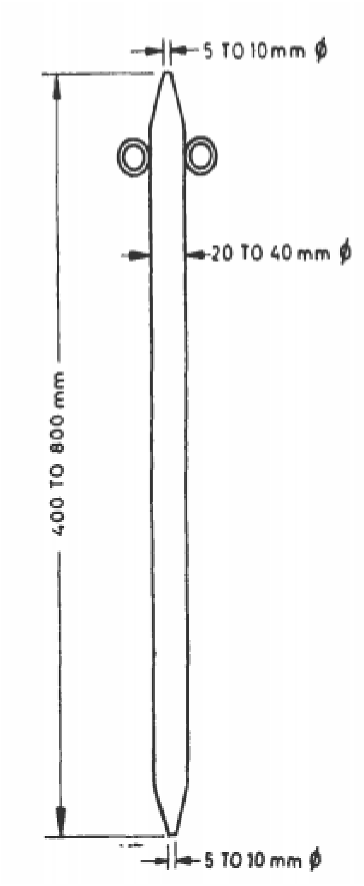

FIG. 1 SAMPLING TUBE

D-2.2 For small containers, the size and shape of the sampling tube may be altered suitably.

D-2.3 In case of sampling from bulk storage tank (through circulation pump sampling valve) suitable sampling container can be used.

D-2.4 In case of filled tanker deeper sampler may be used (sampling from top side) or through sample point arranged to filling line, if tanker/container is under nitrogen pressure (special case)

D-3 SCALE OF SAMPLING

D-3.1 Lot

In any consignment, all the containers of the same size and drawn from the same batch of manufacture In any consignment, all the containers of the same size and drawn from the same batch of manufacture shall constitute a lot. If a consignment is known to consist of different batches of manufacture or of different sizes of containers, the containers belonging to the same batch and size shall be grouped together and each such group shall constitute a separate lot.

D-3.2 For ascertaining the conformity of the material in a lot to the requirements of the specification, tests shall he carried out for each lot separately. The number of containers to be selected for this purpose from a lot shall depend on the size of the lot and shall be in accordance with the col 1 and 2 of Table 2.

Table 2 Scale of Sampling
(*Clause* D-3.2)

Lot Size *N* (1)	**No. of Containers to be Selected** *n* (2)
Up to 3	*
4 to 15	3
16 to 40	4
41 to 65	5
66 to 110	7
111 to 180	8
181 to 300	9
301 and above	10

*****When the lot size is 3 or less, the number of containers to be selected and the method of judging the conformity of the lot to the specification requirements shall be as agreed between the purchaser and the supplier.

D-3.3 These containers shall be selected at random from the lot and in older to ensure randomness of selection a random number table as agreed to between the purchaser and the supplier shall be used. In case such a Table 1, not available, the following procedure is recommended for use:

Arrange all the containers in the lot in a systematic manner and starting from any container, count them as 1, 2, …. upto r and so on, where r is the integral part of *N/n* (*N* being the lot size and *n* number of containers to be selected). Every rth container thus counted shall be withdrawn to give a sample for tests.

D-4 PREPARATION OF TEST SAMPLES

D-4.1 From each of the containers selected according to **D-3.3**, a small representative portion of the material sufficient for carrying out tests as indicated in **4** shall be drawn with the help of the sampling tube (*see* **D-2.1**).

D-4.2 Out of these portions, equal quantity of material shall be taken and mixed thoroughly to form a composite sample, not less than 1 500 ml. The composite test sample shall be divided into three equal parts, one for the purchaser, another for the supplier and the third to be used as a referee sample.

D-4.3 The composite samples shall be transferred to containers of 600 ml capacity and shall be sealed and marked with full identification particulars given in **D-1.7**.

D-4.4 The referee test sample shall also bear the seal of both the purchaser and the supplier. It shall be kept at a place agreed to between the purchaser and the supplier, to be used in the case of any dispute between the two.

ANNEX E
(*Foreword*)

COMMITTEE COMPOSITION
Organic Chemicals, Alcohols and Allied Products Sectional Committee, PCD 09

Organization	*Representative(s)*
Chemical Engineering and Process Development Division, NCL, Pune	Dr C. V. Rode (***Chairperson***)
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All India Alcohol-Based Industries Development Association (AABIDA), Mumbai	Shri K. L. Raphael Shri Kirti Gajjar (*Alternate*)
All India Distillers Association (AIDA), New Delhi	Shri V. N. Raina
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Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

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